

I. Introduction

In order to investigate and quantify the gas phase concentration of benzene evolved from an evaporating pool of both the neat compound and a complex hydrocarbon mixture containing benzene, direct sampling mass spectrometer was employed, sampling downstream from the evaporating benzene or mixture. The direct sampling mass spectrometer offers a distinct advantage over other more traditional gas phase sampling methodologies with respect to the frequency at which benzene concentration can be obtained. Using no chromatographic separation and simply leaking a small amount of sample into the vacuum chamber of the mass spectrometer to be ionized with the resulting ions separated by mass to charge ratio, a direct sampling mass spectrometer can deliver data at up to 10 Hz. Traditional gas phase methods for the detection and quantification of benzene rely on chromatographic techniques to separate benzene from other compounds in the air, and the acquisition of one benzene concentration data point may take more than 30 minutes. Moreover, collection media are often involved, requiring the user to sample air over or into the media and forward the collected sample to a separate laboratory for analysis. Because the parameters of the presently discussed study would dictate that the benzene would evaporate in a matter of minutes and the direct sampling mass spectrometer allows for the real-time collection of benzene concentration data, the technique was deemed appropriate and necessary. Although not accomplished in this study, the method could potentially allow for the quantitative interrogation of the other compounds present in a benzene-containing mixture as well.

The direct sampling mass spectrometer chosen for this work was the ICx Analytical Instruments' ChemSense 600. After-market options exist that can add direct sampling capabilities to other mass spectrometers, but the ChemSense is the only commercially available mass spectrometer specifically designed as a direct sampling unit. Optimized for the automated detection of chemical warfare agents and toxic industrial compounds, the ChemSense is also able to interrogate, quantitatively, gas phase species such as benzene, even in complex hydrocarbon mixtures. Additionally, the author's familiarity with this piece of chemical instrumentation, having helped to design and test pre-production models, contributed to its

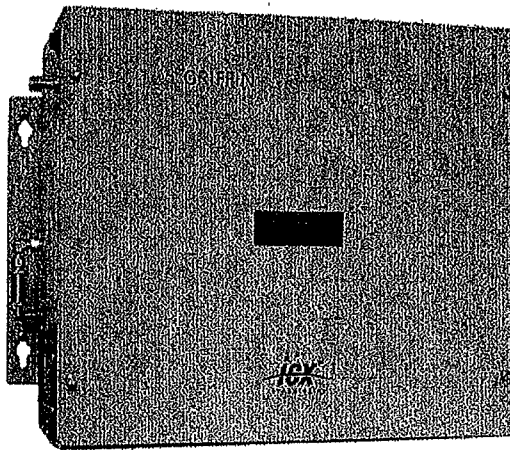
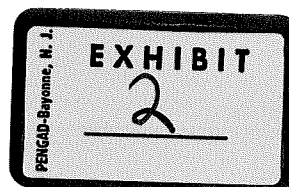


Figure 1. ICx Analytical Instruments' ChemSense 600 Direct Sampling Mass Spectrometer.

selection for this work.



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The ChemSense mass spectrometer uses a cylindrical ion trap mass analyzer¹ (CIT) to separate ions according to their mass-to-charge ratios. Ions are created inside of the CIT by electron ionization. These electrons are generated in a glow discharge source. Neutral sample compounds are admitted to the vacuum chamber, within which the CIT and glow discharge source reside, though either a capillary restriction or upon being thermally desorbed from sorbent media upon which they are preconcentrated during sampling³.

II. Method Development

Initial method development questions to be answered included the question of whether the capillary leak inlet, which would provide data frequencies greater than 1 Hz, or the sorbent tube preconcentration inlet system (providing a data point approximately every 30 s) would provide the best data quality for the benzene evaporation rate study. Both inlet options were initially considered and characterized by exposing the ChemSense to gas phase benzene at various concentrations generated by diluting (to various degrees) a 5 ppm pre-mixed benzene cylinder with zero air (less than 1 ppm hydrocarbons). Figure 2 represents the system used to generate benzene at various gas phase

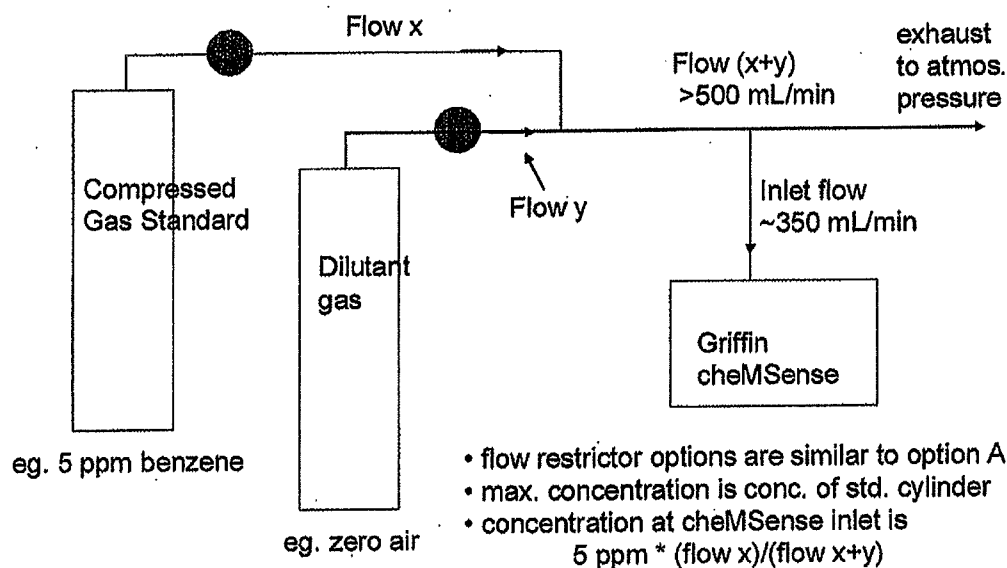


Figure 2. Schematic representation of the inlet characterization and calibration system with performance and limitation notes

concentrations for use in characterizing both potential mass spectrometer inlet modes and in calibrating the mass spectrometer.

Mass spectrometers may be programmed, usually through the control software, to record data in several different manners. The CIT in the ChemSense is capable of performing full scan mass analyses, where the ion signal over a broad mass-to-charge range is recorded continuously. This method of mass analysis has the advantage of collecting data, potentially, for a broad range of chemical compounds that may be present in a mixture. Alternatively, data may only be recorded for ions of a single mass-to-charge ratio, or more advanced techniques, like tandem mass spectrometry, in which ions are intentionally fragmented inside the CIT, may be employed. In order to gain the most information about benzene and the other

compounds present in the mixtures studied in this work, data was recorded over a mass-to-charge range of 50 to 150. This mass-to-charge range includes the dominant ion signals from most of the compounds anticipated to be in the mixtures studied. Other important instrument

Table 1. Important mass spectrometer parameters

Item, setpoint, or other characteristic	Value or notation
Vacuum manifold pressure	$>1.5 \times 10^{-5}$ torr
CIT bath gas	air
Inlet manifold temperature	130 C
Inlet flow rate	360 mL/min
Particulate filter temperature	95 C
Electron multiplier bias	1300 V
CIT temperature	130 C

parameters which may be important for the future replication of this work are included in Table 1.

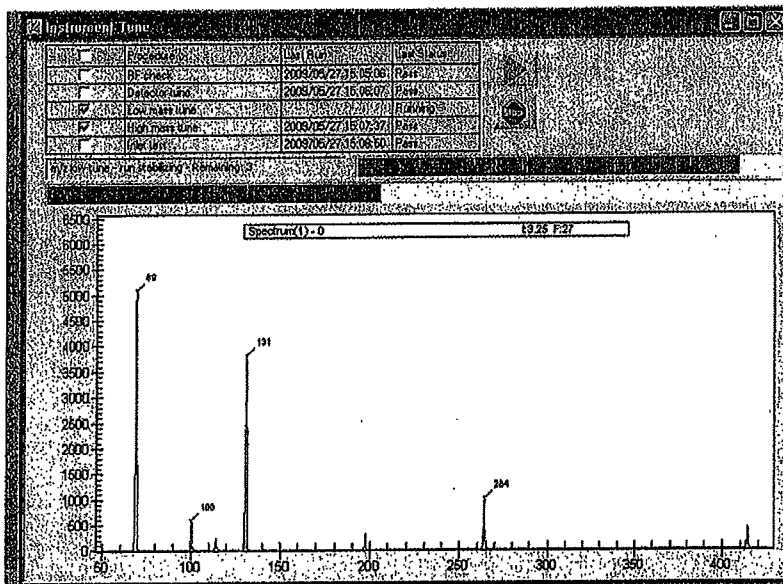


Figure 3. Mass spectrum indicating the mass-to-charge calibration of the ChemSense is correct. This check was performed daily prior to data collection for the day.

Prior to each day's data collection, the ChemSense's mass-to-charge axis was calibrated by running a software controlled tuning procedure called "low m/z tune." Figure 3 shows the resulting mass spectrum of the calibration compound, perfluorotributyl-amine.

II-a. Pre-concentration inlet evaluation

The ChemSense is